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STUDIES OF ALKALI METAL CORROSION ON MATERIALS FOR ADVANCED SPACE POWER SYSTEMS

Quarterly Progress Report No. 4

For Quarter Ending June 26, 1965

By
R.W. HARRISON

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SPACE POWER AND PROPULSION SECTION
MISSILE AND SPACE DIVISION
GENERAL ELECTRIC
CINCINNATI, OHIO 45215

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STUDIES OF ALKALI METAL CORROSION ON MATERIALS FOR ADVANCED SPACE POWER SYSTEMS

QUARTERLY PROGRESS REPORT 4

Covering the Period

March 26, 1965 to June 26, 1965

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I. INTRODUCTION

The program reviewed in this fourth quarterly progress report, covering from March 26, 1965 to June 26, 1965, is sponsored by the National Aeronautics and Space Administration. Its purpose is to examine the influence of stress on the corrosion behavior of an advanced refractory alloy in potassium (Task I) and to investigate corrosion mass transfer effects in a stainless steel-columbium alloy-potassium system (Task II).

Task I

While there is considerable evidence that refractory alloys have excellent corrosion resistance to potassium, there are few experiments which describe the possible effects of stress on corrosion when the stress is sufficiently large to produce substantial amounts of creep during the test. It is appropriate for comparative purposes to study an advanced refractory alloy which has demonstrated excellent corrosion resistance to refluxing potassium during long-time exposures conducted at relatively low stresses at 2000°F. In this regard, D-43 columbium base alloy, in the form of welded capsules, has been tested in potassium under refluxing conditions for periods of 5,000 and 10,000 hours at temperatures on the order of 2000°F[1] and has been selected for inclusion in this program.

The D-43 alloy reflux capsules shall be tested under conditions which result in about 5 to 10% strain during a 500- to 2,000-hour exposure period in the 2000° to 2200°F temperature range. The reflux capsules used in this study will be of similar size to those previously described (Ref. 1). The capsule wall shall be reduced in the potassium liquid region and in the vapor condensing region to provide gauge sections where the extent of creep can be measured. Moderate temperature adjustments can be made during the test to achieve the desired strain-time conditions.

Task II

The use of stainless steel, rather than refractory alloys, for power plant radiator construction and for the lower temperature portion of experimental facilities constitutes material and fabrication cost savings. Two methods of employing this approach are: use of co-extruded, stainless steel shell-refractory alloy core, tubing in the radiator or use of an all stainless steel radiator joined to the system by a bimetallic joint. Although the latter approach is preferred considering cost and problems associated with fabrication and joining of co-extruded tubing, a major uncertainity and limitation arises from the mass transfer of interstitial elements from the stainless steel to the refractory alloys through the alkali metal.

It is well established that the carbon and nitrogen transfer from Type 316SS to Cb-1Zr alloy at temperatures near 1500°F (Ref. 2). While some important aspects of this mass transfer behavior have been examined, several critical details require additional investigation. There is a need to define acceptable time and temperature conditions of operation in terms of maintaining satisfactory performance of the refractory alloys, such as Cb-1Zr alloy. Also, there are certain metallurgical aspects of this behavior which should be investigated in an effort to eliminate or reduce the mass transfer rate. In the latter category, it is most appropriate to consider the stabilization of carbon and nitrogen in the stainless steel by the addition of metallic elements which form carbides and nitrides of high thermodynamic stability. Commercially available, titanium stabilized, Type 321SS is one such alloy. A comparative investigation of this alloy and Type 316SS was performed to indicate the ability of the titanium addition to reduce or eliminate interstitial mass transfer in a stainless steel-Cb-1Zr alloy bimetallic system. Columbium-1% zirconium alloy specimens were exposed to liquid potassium in Type 321SS and Type 316SS capsules for 1,000 hours at 1400°F under isothermal conditions to evaluate this premise. Post-test evaluation showed that Type 321SS has a significant advantage over Type 316SS in refractory metal-stainless steel-potassium systems in inhibiting mass transfer of the interstitial elements carbon and nitrogen from the stainless steel to the refractory metal.

II. SUMMARY

During the fourth quarter of this program, the topics abstracted below were covered. The results are interpretatively presented in this report.

Task I - Stress Corrosion Reflux Capsule Tests

A preliminary stress corrosion reflux capsule test was terminated as a result of excessive expansion in the reduced wall sections of the capsule during the heat-up cycle. In subsequent investigations, an instrumentation error was found in the thermocouple circuitry resulting in a positive temperature error of approximately 400°F. This instrumentation error has been corrected, and another capsule has been placed on test, as described below.

Two additional D-43 alloy stress corrosion reflux capsules were fabricated and heat treated for 1 hour at 2400°F. One capsule was filled with potassium and installed in the test facility.

A split tantalum strip heater for the boiling nucleator was designed, constructed, and installed in the test facility.

The capsule test has been initiated. Creep rate measurements indicated a faster rate at 2250°F than calculated from pretest uniaxial creep data. After additional temperature adjustements and creep rate measurements, a temperature of 2100°F was selected to give the desired creep strain (5-10%) in the time duration specified

(500-2,000 hours). To date, 430 hours of test time has been accumulated, and the creep strain has reached 2 to 3%.

Task II - Bimetallic Isothermal Capsule Tests

Post-test evaluation has been completed.

Preparation of a topical report has been initiated.

III. TASK I - STRESS CORROSION REFLUX CAPSULE TESTS

Preliminary Capsule Test

A. Test Facility

The initiation of the preliminary capsule test was postponed pending receipt of tungsten caps for the Al_2O_3 probes. The machined tungsten caps were received and installed on the Al_2O_3 probes, Figure 1. Subsequently, the LVDT-probe units were reassembled, Figure 2, and installed in the test facility insitu, Figure 3. The system was closed and evacuated. Mass spectrometer leak checking indicated no leaks present and a pre-bakeout vacuum of 2×10^{-8} torr was attained. The system was baked out at 350°C for 8 hours; on cooling, a vacuum of 1.5×10^{-9} torr was measured with a tubular Bayard-Alpert ionization gauge.

B. Capsule Testing

The instrumentation checkout indicated no apparent problems and heat-up of the capsule was initiated. The temperature of the capsule was increased at a rate to maintain a chamber pressure below 1×10^{-6} torr. At a measured liquid zone temperature of approximately 1100°F, boiling instability induced capsule vibration which was indicated on the LVDT recorder. The boiling nucleator was activated, and when the temperature of the boiling nucleator was increased to 300°F above that measured in the capsule liquid zone,

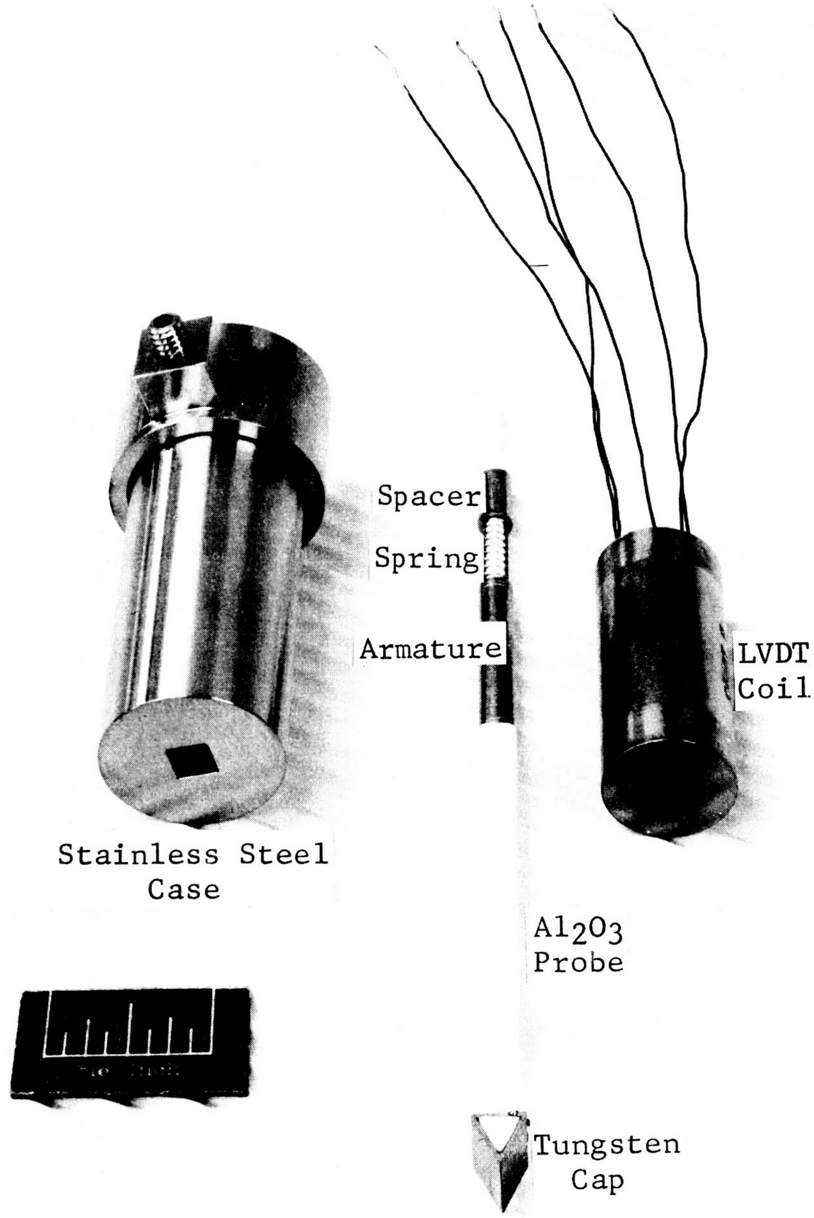

Figure 1. Disassembled Strain Measuring LVDT-Probe Unit with Tungsten Cap Installed.

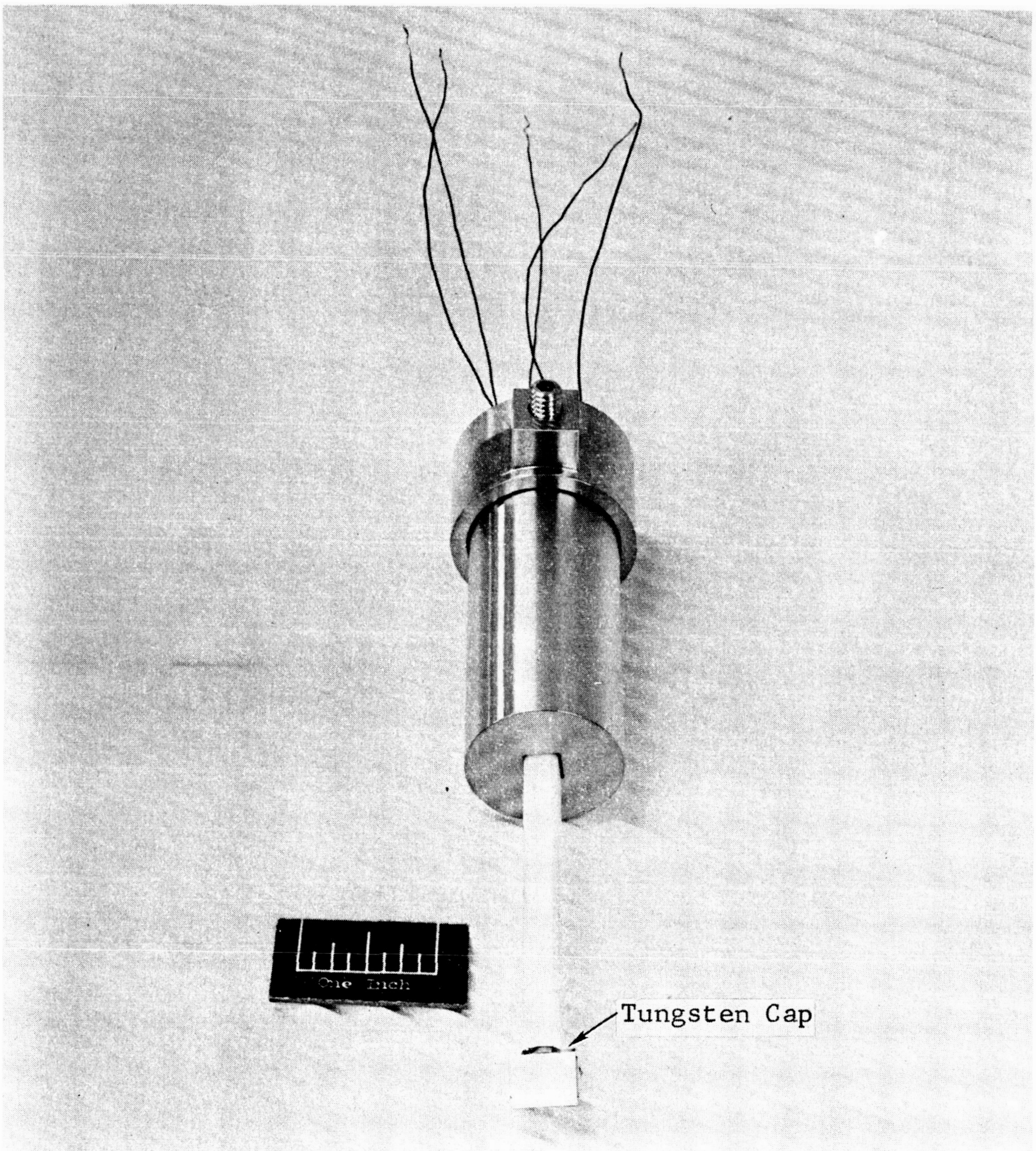

Figure 2. Assembled LVDT-Probe Unit with Tungsten Cap.

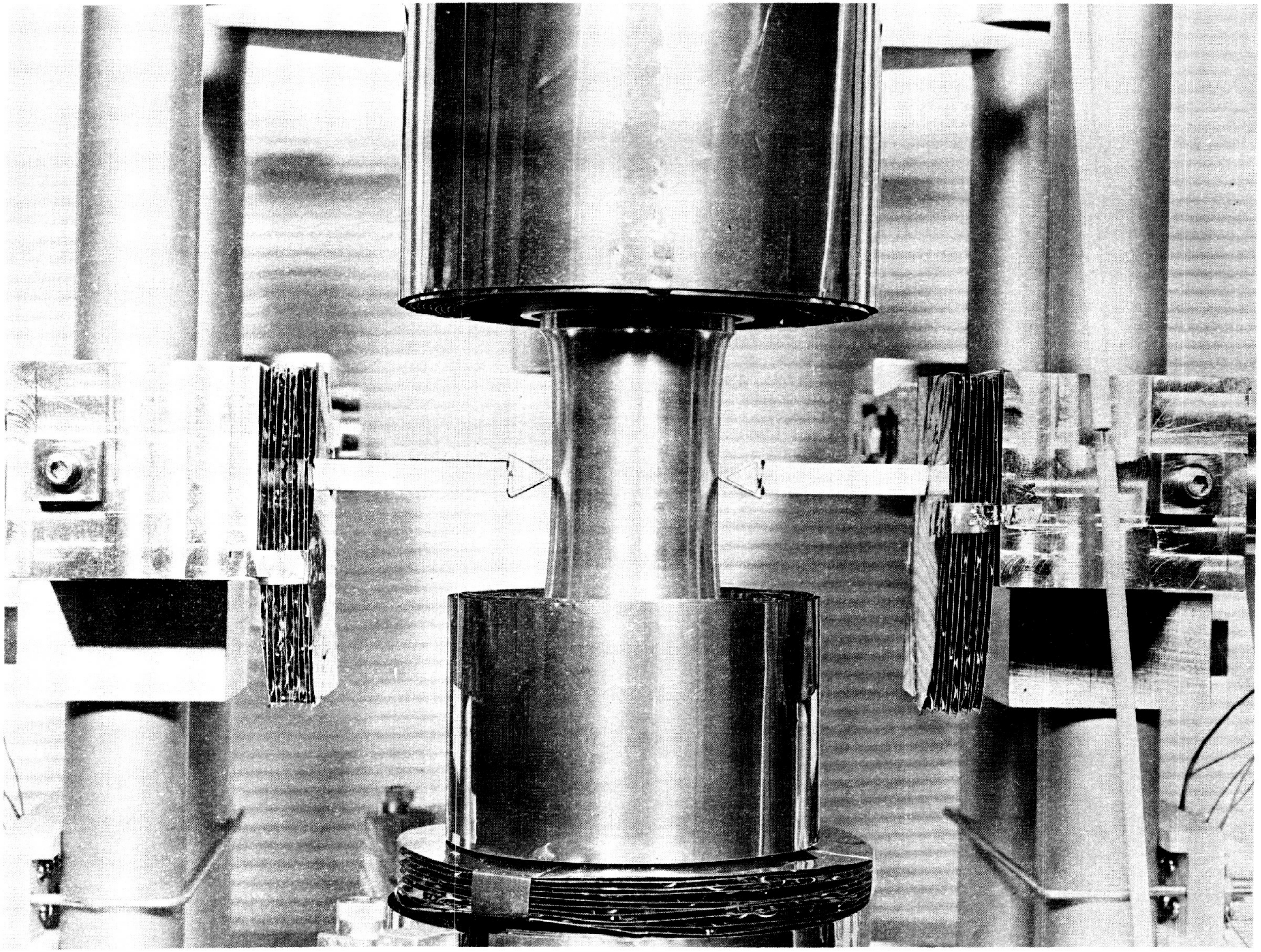

Figure 3. Tungsten Capped LVDT-Probe Units Installed in Vapor Region of Capsule Test Facility.

the capsule vibration ceased and the previously measured 100°F temperature differential between the liquid and vapor zone dropped to within 15°F, Figure 4. The advantages of such a boiling nucleator in preventing the possibility of damage to sensitive components resulting from capsule vibration is thus evident. By maintaining the boiling nucleator temperature approximately 300°F above the temperature of the potassium liquid as heating of the capsule was continued, stable boiling was maintained.

However, at a measured temperature of 1980°F, the boiling nucleator heater shorted out causing a pressure rise that necessitated a temporary shutdown of the test.

The heater for the boiling nucleator was replaced with a similar heater and the system was closed, evacuated and baked out. A cold wall pressure of 1.5×10^{-9} torr was attained prior to restart with similar start-up procedures being employed in the second heat-up as were employed previously. At a measured temperature of 2035°F rapid expansion of the capsule wall was observed on the LVDT recorder and by visual examination through the sightport. The current to the heaters was turned off immediately and testing was terminated. The degree of expansion in the capsule liquid and vapor regions is shown in Figures 5 through 8. Subsequent checking of the possibilities capable of producing such an anomally revealed

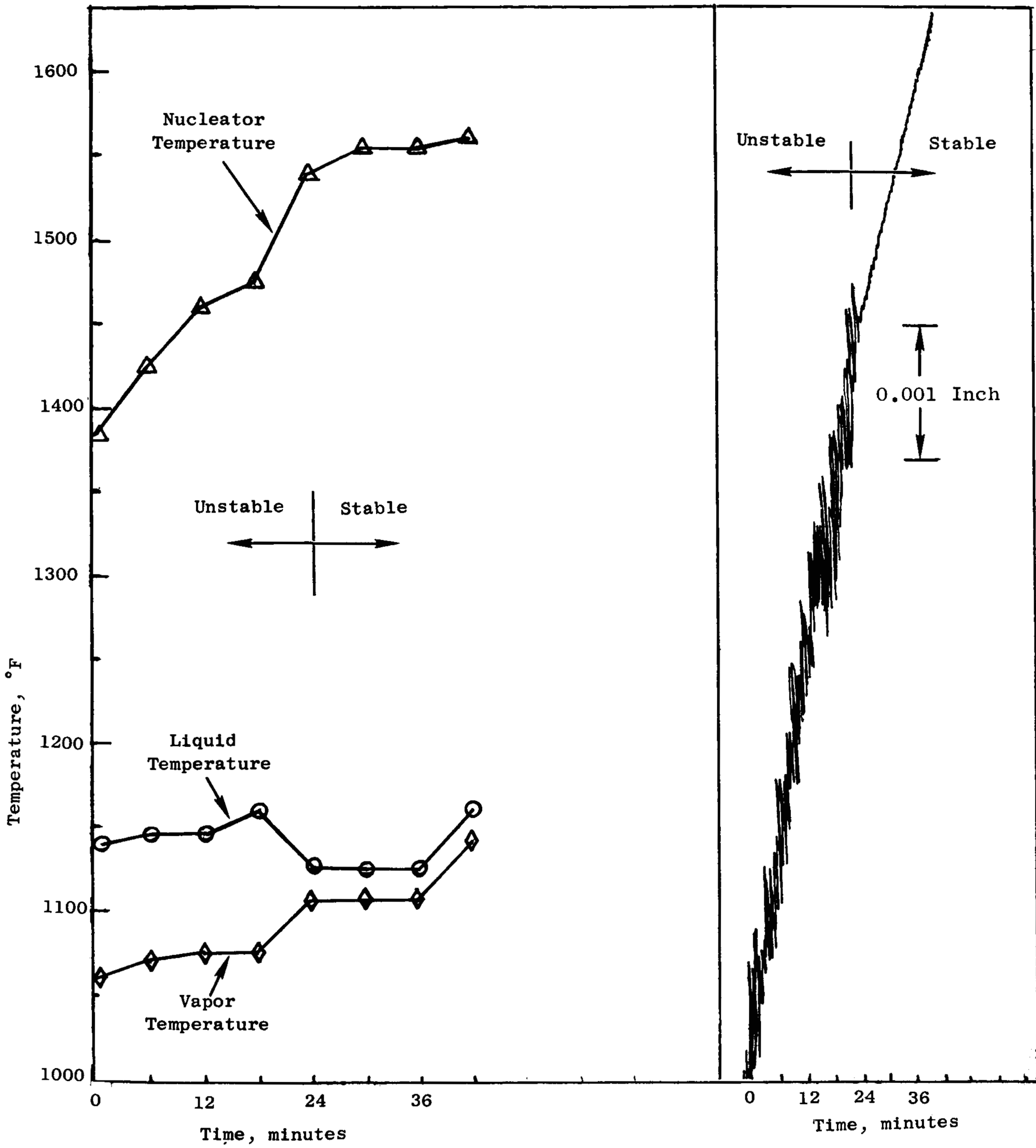

Figure 4. Effect of Boiling Nucleator Temperature on Boiling Instability and Induced Vibration in a Refluxing Potassium Capsule.

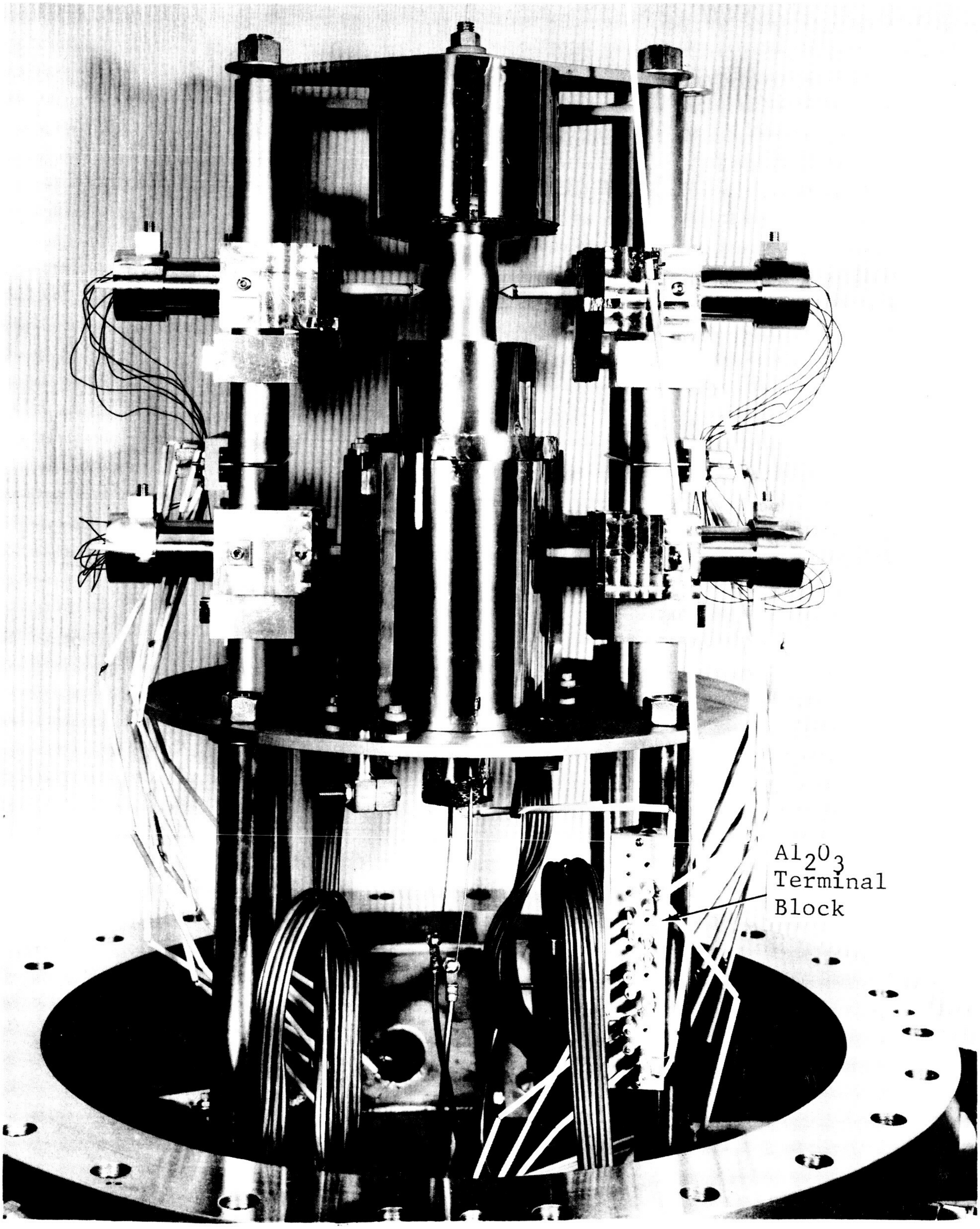

Figure 5. Test Facility Showing Expansion of the Capsule Wall in the Condensing Zone of the D-43 Alloy Preliminary Capsule Test.

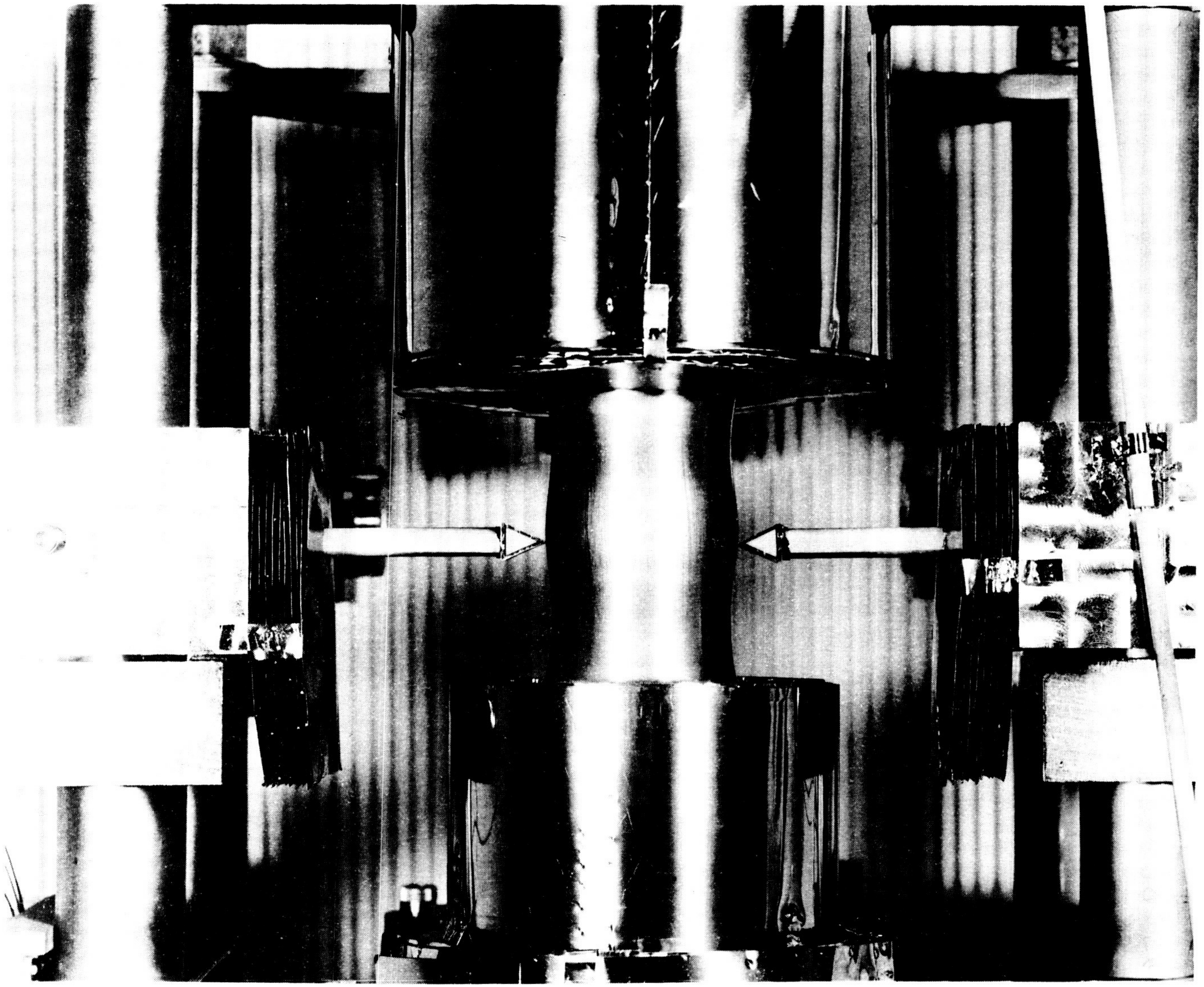

Figure 6. View of Condenser Region of the Preliminary D-43 Alloy Stress-Corrosion Capsule Showing the Extent of Expansion that Occurred During Test.

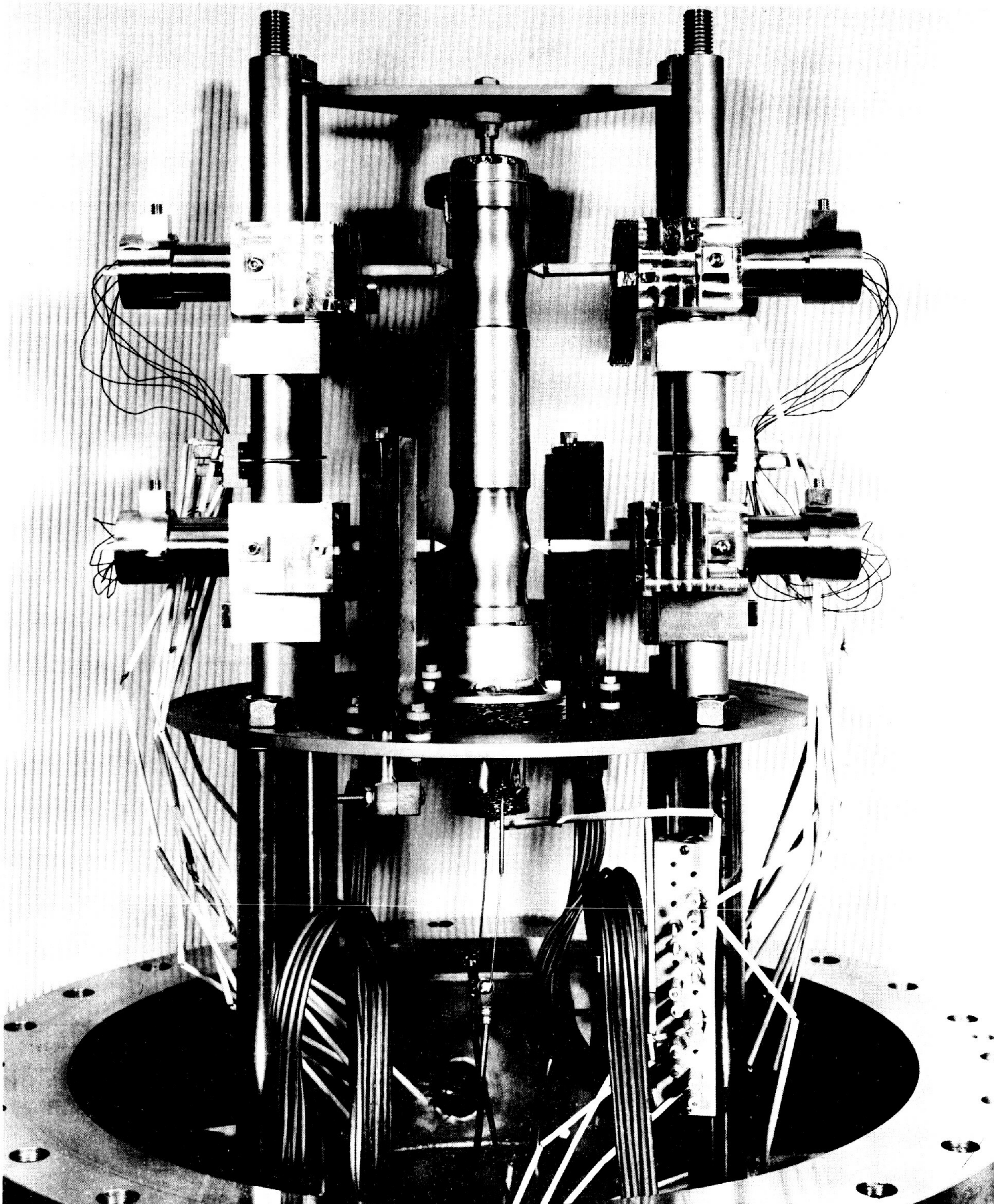

Figure 7. Test Facility Showing the Expansion that Occurred in the Condenser and Liquid Regions of the D-43 Alloy Preliminary Capsule Test.

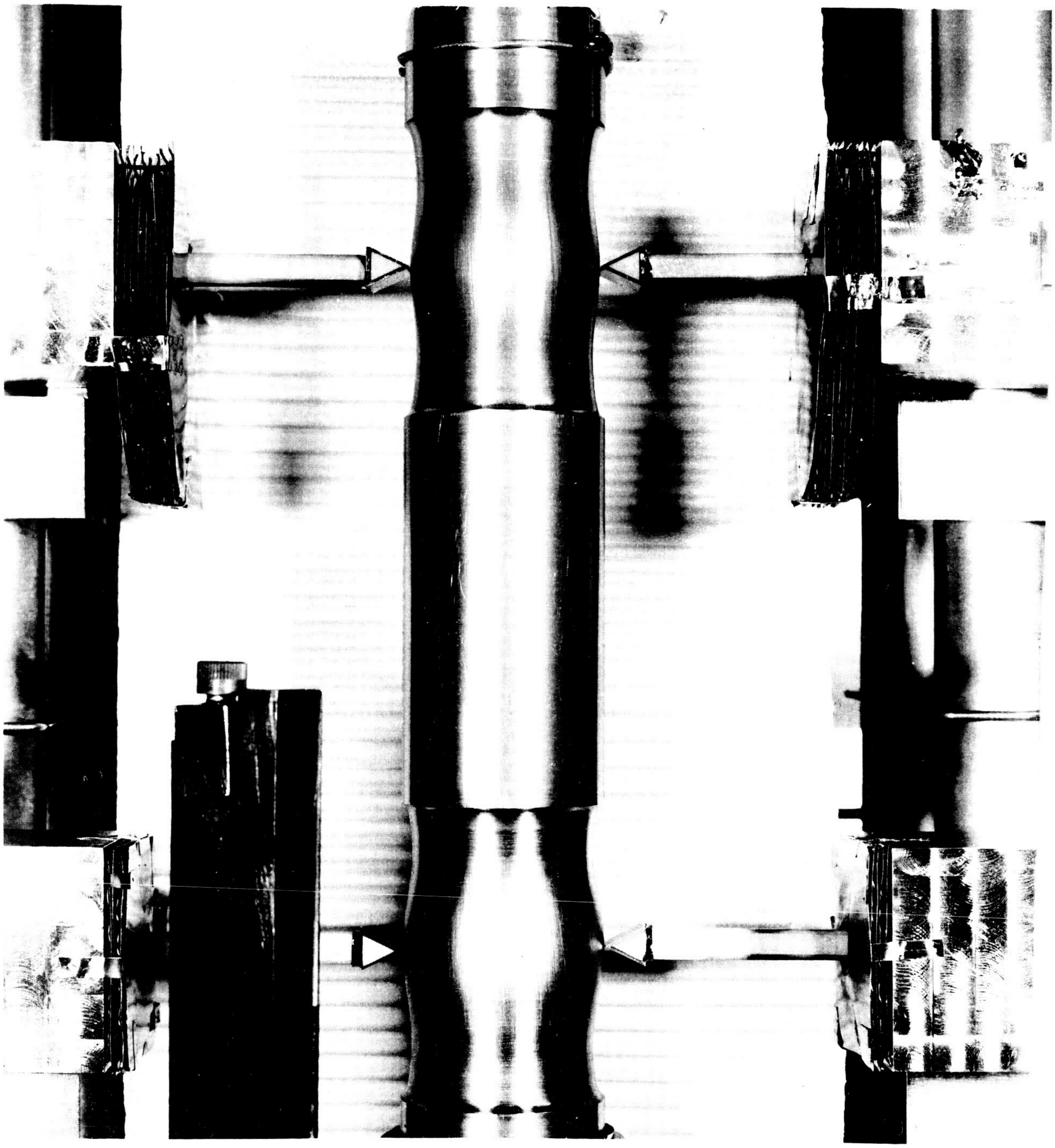

Figure 8. View of Condenser and Liquid Regions of the D-43 Alloy Preliminary Stress-Corrosion Capsule Showing the Extent of Expansion that Occurred During Test.

an error in the instrumentation of the W+25%Re vs W+3%Re thermocouples. The W+25%Re vacuum feedthrough wires were connected to the W+3%Re legs of each thermocouple and the W+3%Re vacuum feedthroughs wires were connected to the W+25%Re legs of each thermocouple. This introduces two thermocouple junctions at the Al_2O_3 terminal block shown in Figure 5. The induced mv output from these two junctions must be added to the mv output from the primary thermocouple junction in order to obtain the correct temperature. If it is assumed that the temperature of the Al_2O_3 terminal block was 300°F, the corrected temperature of the capsule would be 2450°F when the measured mv output indicated a temperature of 2035°F. At a temperature of 2450°F, the observed behavior of the capsule would be consistent with the measured creep properties of the D-43 alloy bar from which the capsule was fabricated[3].

The D-43 alloy capsule has been opened under argon and the potassium has been drained. Subsequently, the capsule was cleaned by vacuum distillation and sectioned, Figure 9.

On the basis of the estimated capsule temperature, the wall expansion, Figure 10, corresponds to 16% creep strain in the liquid region and 12.5% creep strain in the condensing region. Lüders lines (stretcher strains), from the localized plastic deformation in the reduced capsule wall were evidenced. Different surface patterns

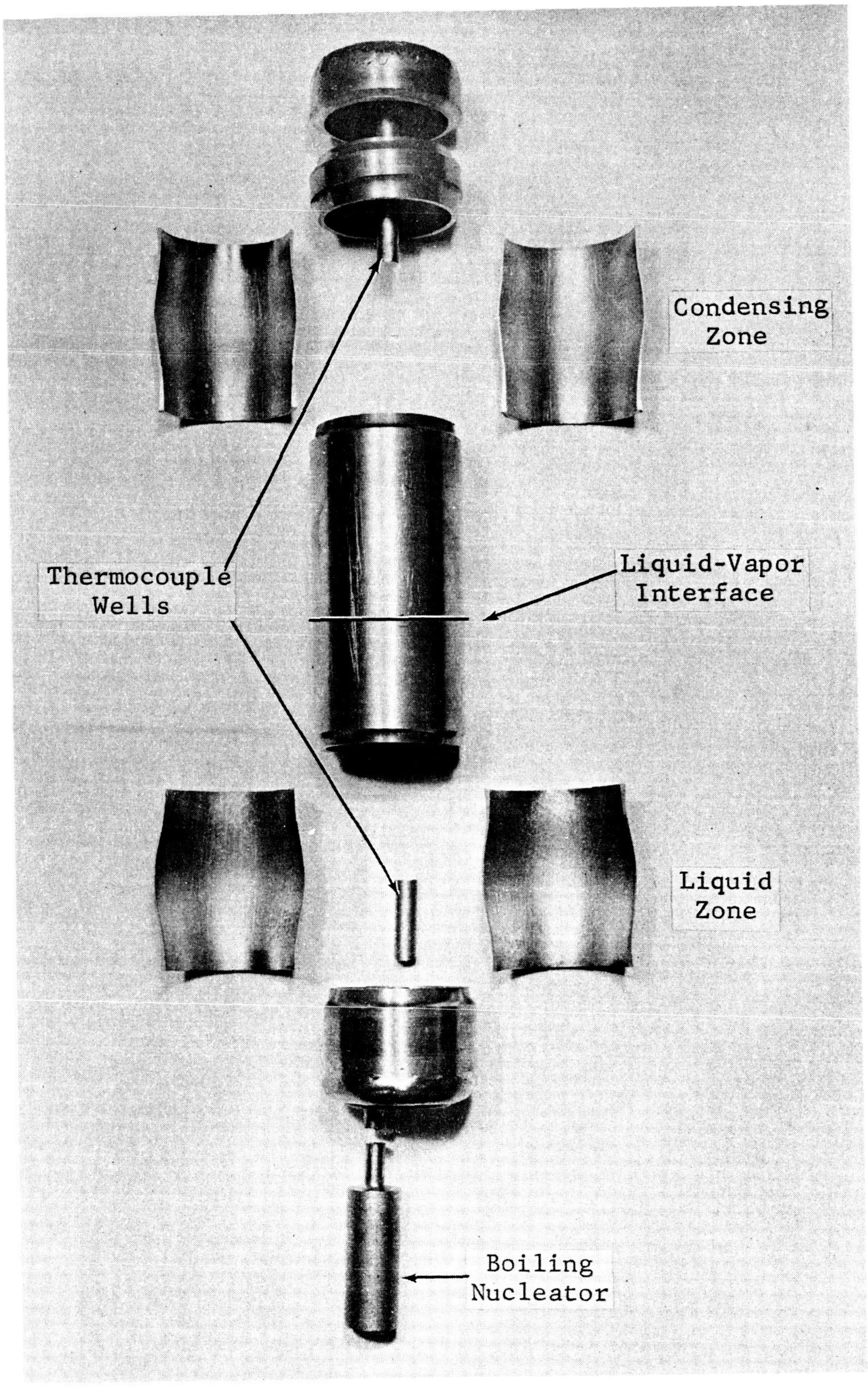

Figure 9. D-43 Alloy Preliminary Capsule Test Sectioned. Test Terminated on Heat-Up as a Result of Extensive Expansion in the Liquid and Condensing Zone.

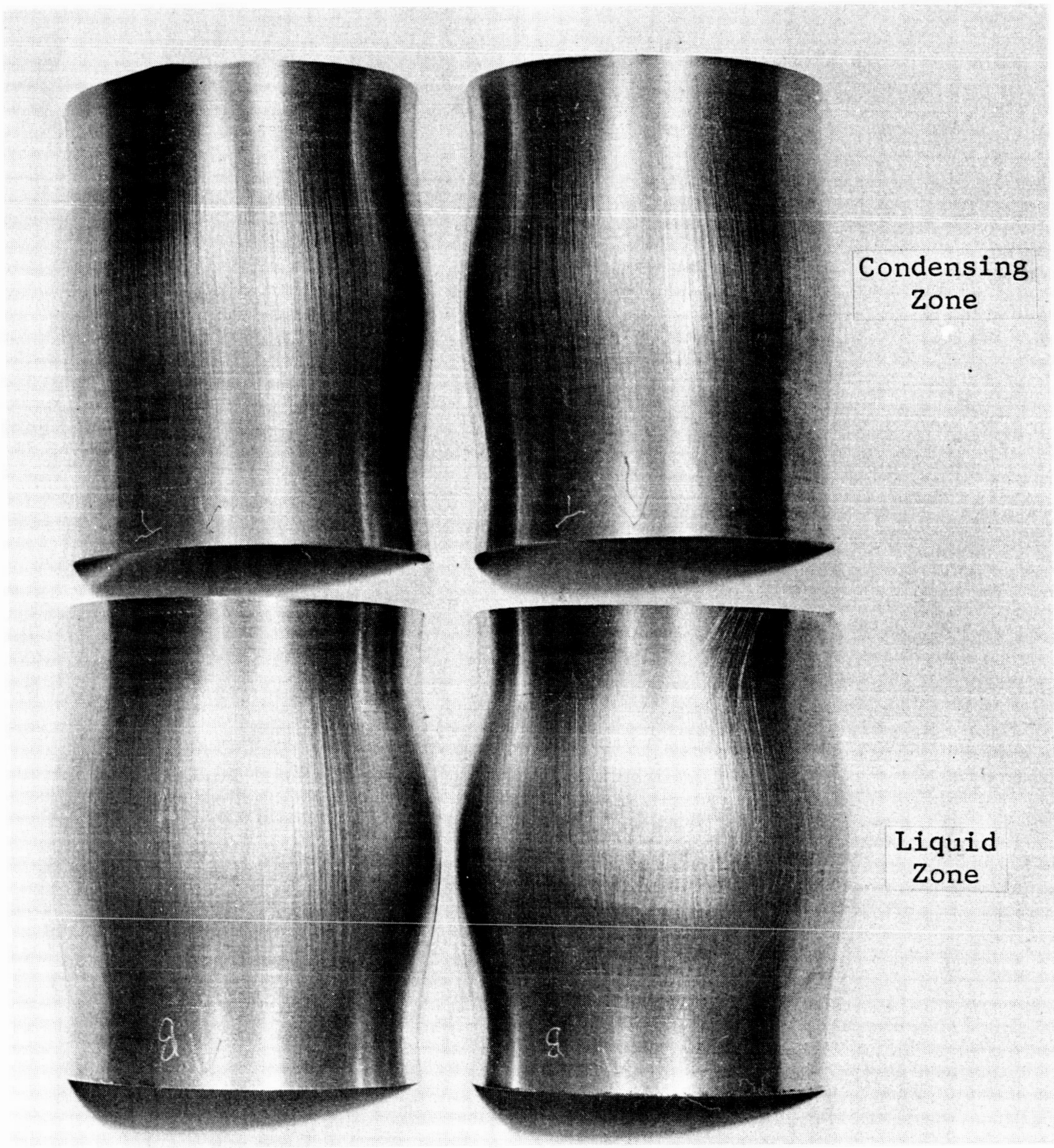

Figure 10. Sectioned Liquid and Condensing Zones From D-43 Alloy Preliminary Capsule Test (Outside Diameter).

were observed in the liquid and condensing zones of the capsule as a result of potassium exposure, Figure 11. The pattern change was found to occur at the liquid interface away from the gauge sections, therefore, is not stress dependent.

Macrophotographic examination of the liquid region at 30X magnification did not delineate this pattern; however, the spots in the condensing region were observed.

One of the series of elongated spots running horizontally to the capsule axis in the condensing region shown previously in Figure 11, is shown at higher magnification in Figure 12. A series of vertical spots along scratches are depicted in Figure 13. Metallographic examination of the surface cross section at these spots indicated no apparent structure or carbide morphology change, and the maximum depths were less than 0.001 inch.

D-43 Alloy Stress Corrosion Reflux Capsule Test I

A. Capsule Fabrication and Filling

A second D-43 alloy reflux capsule with a boiling nucleator was fabricated, leak checked, and the welds radiographed. In compliance with a request from the NASA Technical Manager, the capsule was postweld annealed for one hour at 2400°F in a vacuum of 10^{-6} torr. The capsule was wrapped in Cb-1Zr alloy foil on the inside and on the outside prior to heat treatment, Figure 14. Although a

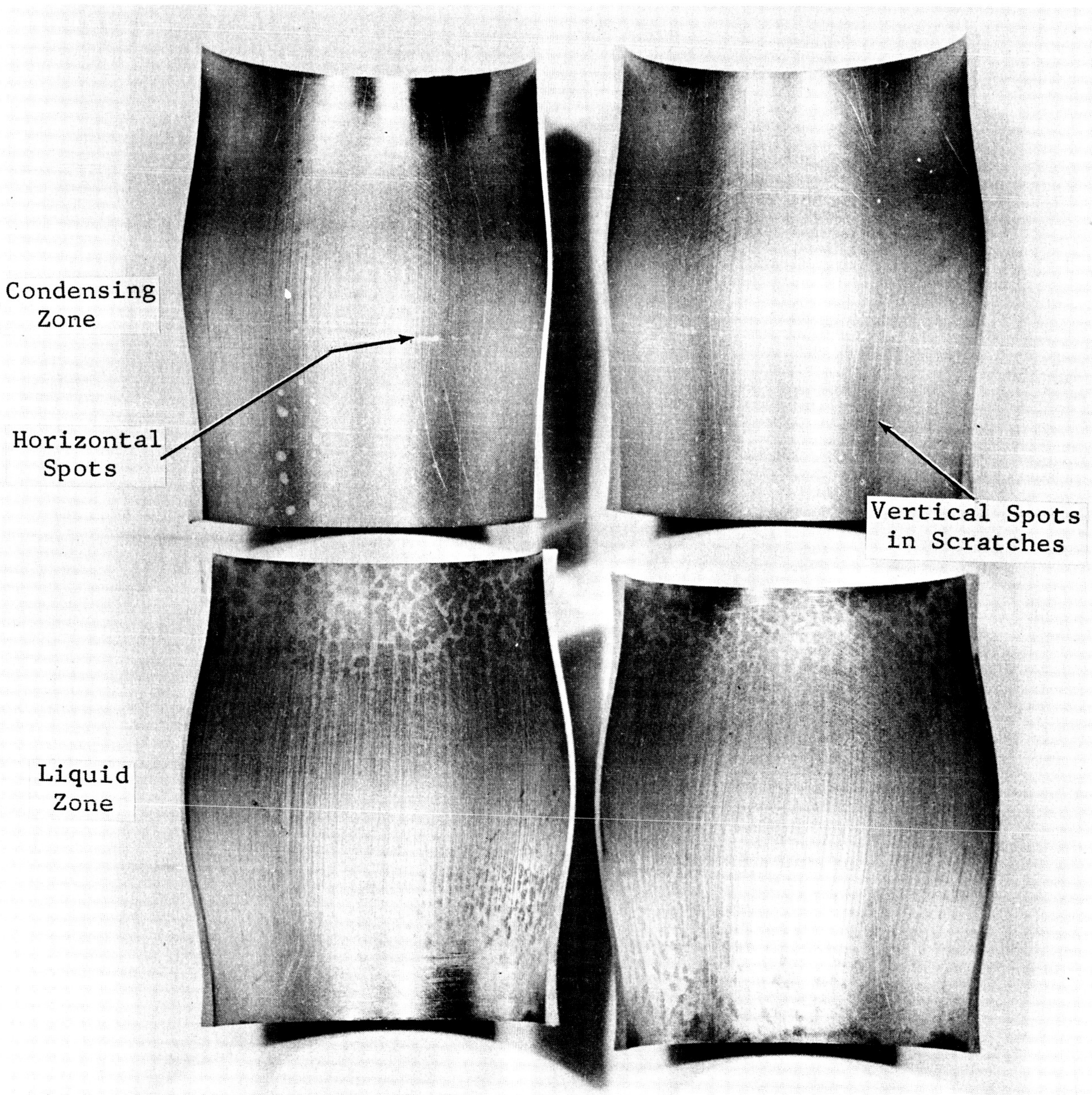

Figure 11. Sectioned Liquid and Condensing Zones from D-43 Alloy Preliminary Capsule Test (Inside Diameter) Showing Surface Patterns Produced by Potassium Exposure.

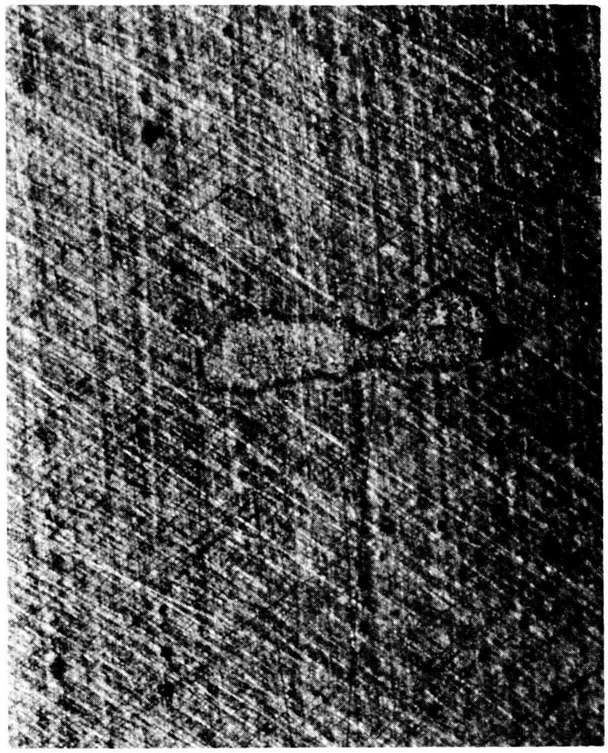

Figure 12. Horizontal Spot in Condensing Zone of D-43 Alloy Preliminary Capsule Test Exposed to Potassium Vapor at an Estimated Temperature of 2450°F. Mag: 30X

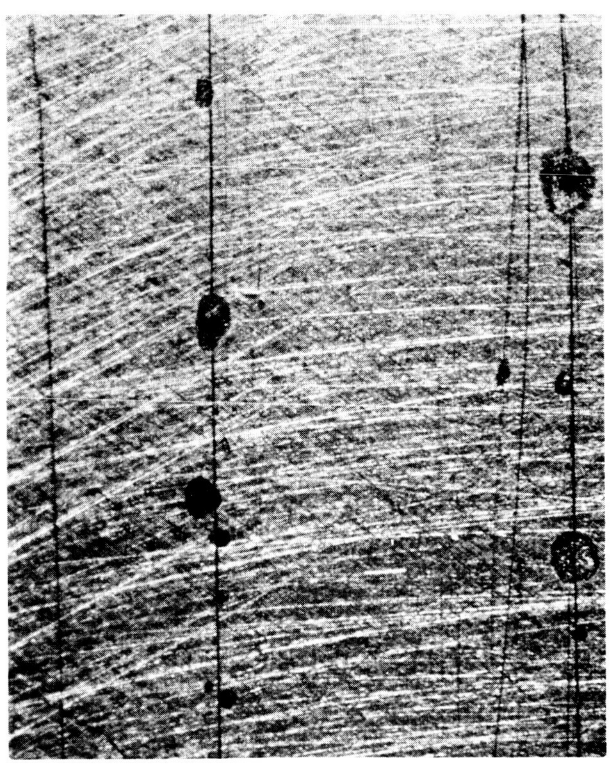

Figure 13. Vertical Spots Along Scratches in Condensing Zone of D-43 Alloy Preliminary Capsule Test Exposed to Potassium Vapor at an Estimated Temperature of 2450°F. Mag: 30X

Figure 14. D-43 Alloy Stress Corrosion Reflux Capsule Wrapped in Cb-1Zr Alloy Foil for Heat Treatment Following Final Machining.

Cb-1Zr alloy control specimen was heat treated along with the capsule, post-test analysis for possible contamination was not warranted because the vacuum was maintained in the 10^{-6} torr range. Subsequently, the capsule was filled with potassium to produce a 5-inch liquid height at the projected test temperature (approximately 2250°F). The potassium was transferred directly to the capsule from the final hot trapping container, and the capsule was sealed by electron beam welding in a vacuum of 1×10^{-5} torr. The potassium that was used for filling the capsule was sampled at the same time the capsule was filled and analyzed for oxygen by the mercury amalgamation method; the results showed the oxygen in the potassium sample taken from the fill tube to be 5 ppm and the oxygen in the potassium sample that was cast inside the weld chamber to be 12 ppm. The filled and sealed capsule was examined radiographically to assure a sound electron beam weld and subsequently was installed in the test facility.

B. Test Facility

A split tantalum strip heater has been designed and constructed and will be employed in heating the boiling nucleator in this capsule test. This change in design results from the improved reliability of this type heater as compared to that of the tantalum-sheathed resistance element used in the preliminary capsule test. The installation of this heater with appropriate OFHC copper bus bars and 0.003-inch thick tantalum foil shielding is depicted in Figures 15, 16 and 17.

C. Capsule Testing

Subsequent to LVDT calibration and a thorough instrumentation check of the W+3%Re vs W+25%Re thermocouples, the system was closed and evacuated. Mass spectrometer leak checking indicated no leaks present and a pre-bakeout vacuum of 5×10^{-8} torr was attained. The system was baked out at 350°C for 6 hours; on cooling, a vacuum of 2×10^{-9} torr was measured with a tubular Bayard-Alpert ionization gauge.

Heat-up was initiated on 6-7-65. The temperature of the capsule was increased slowly to maintain the pressure in the 10^{-7} torr range. A boiling nucleator temperature of 300°F above that of the capsule was maintained. Similar results of boiling stability where evidenced as described under the Preliminary Capsule Test. At a 1900°F capsule temperature, the boiling nucleator temperature was lowered to that of the capsule. Boiling remained stable with a measured ΔT of 15°F between the liquid and condensing regions. The capsule and boiling nucleator temperatures were increased to 2250°F and stabilized. The measured expansion from the LVDT probe units on the capsule reduced wall sections indicated a faster creep rate at 2250°F than

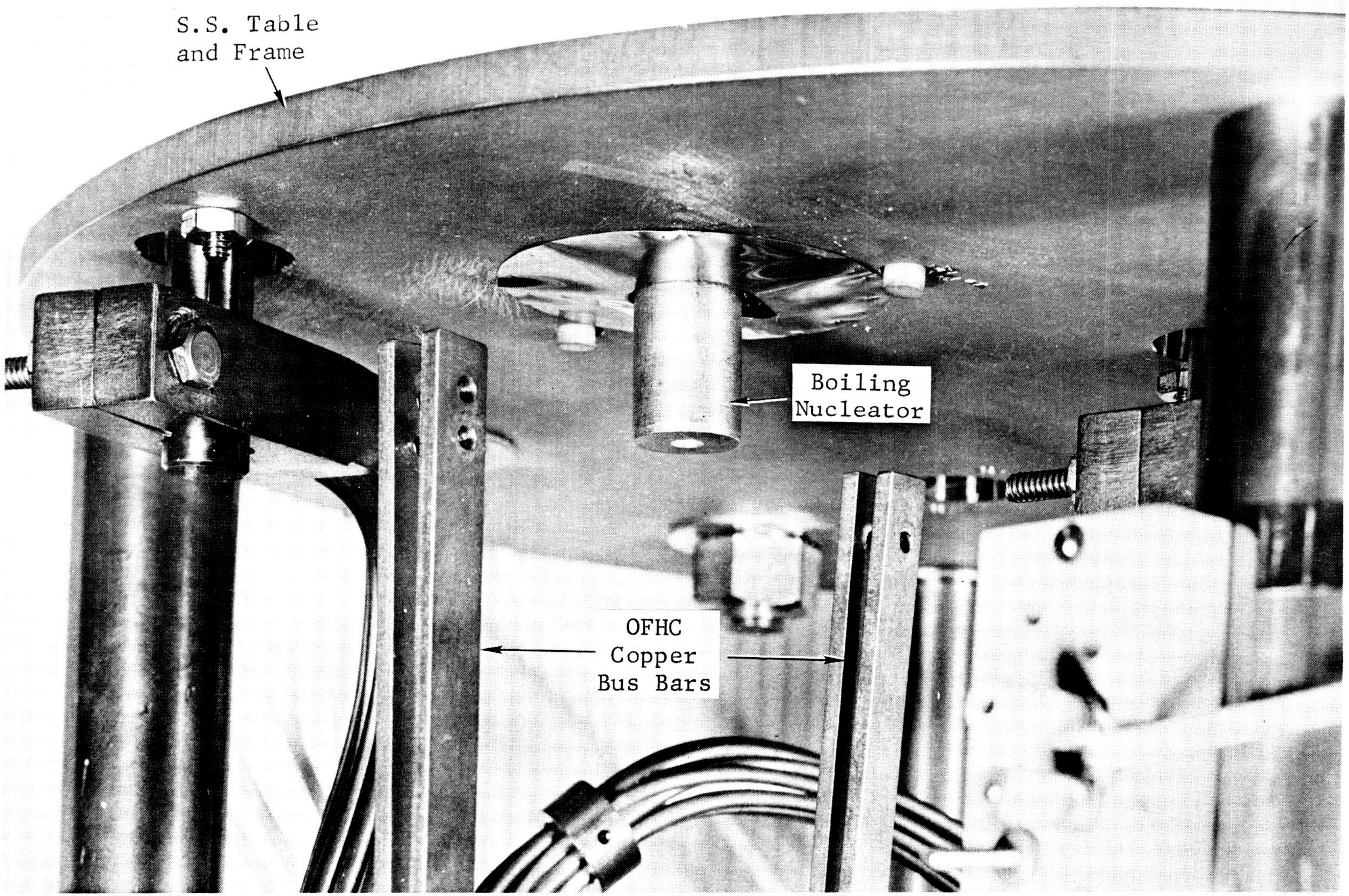

Figure 15. Boiling Nucleator Location in Stress Corrosion Capsule Test Facility.

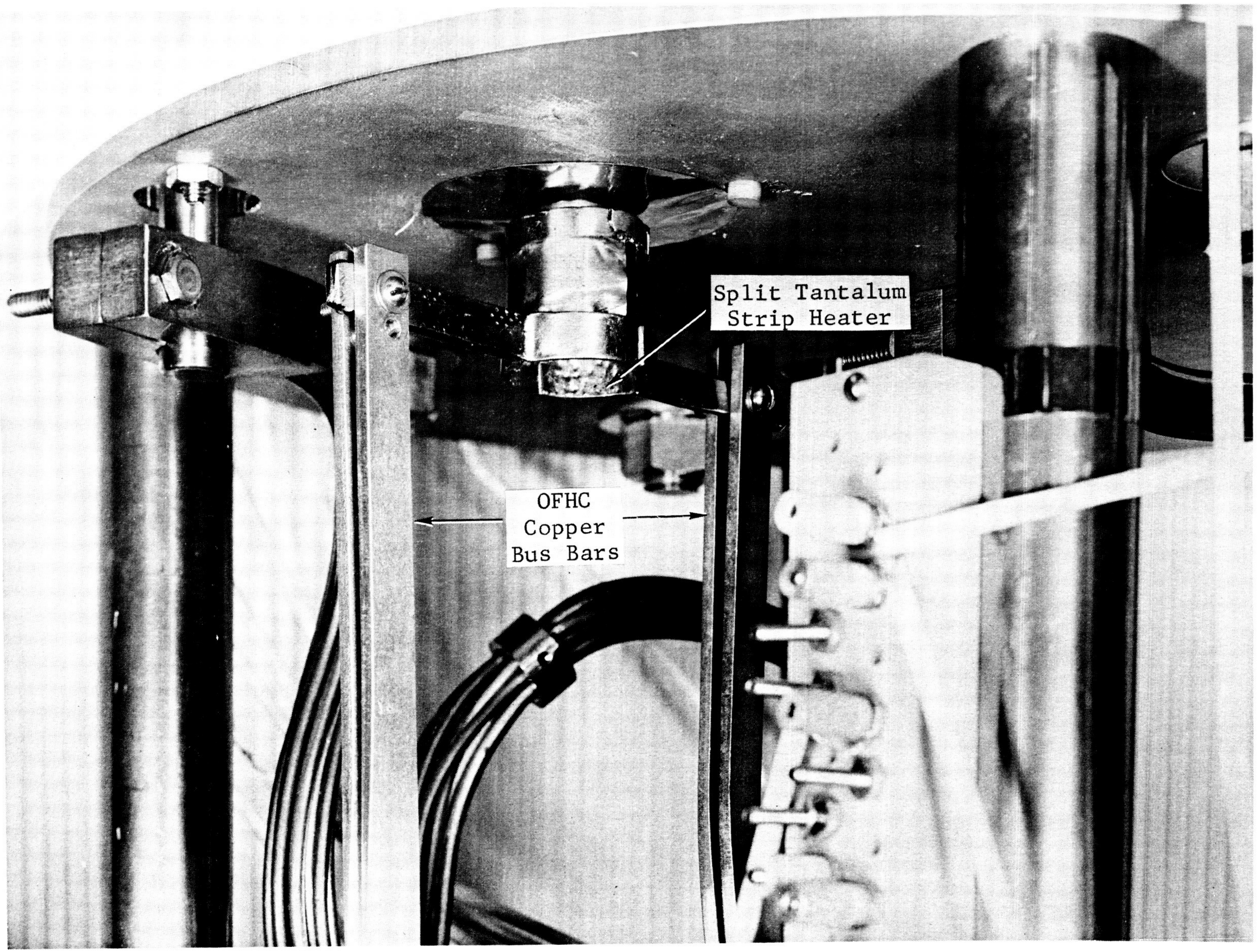

Figure 16. Split Tantalum Strip Heater for Boiling Nucleator Installed in Stress Corrosion Capsule Test Facility.

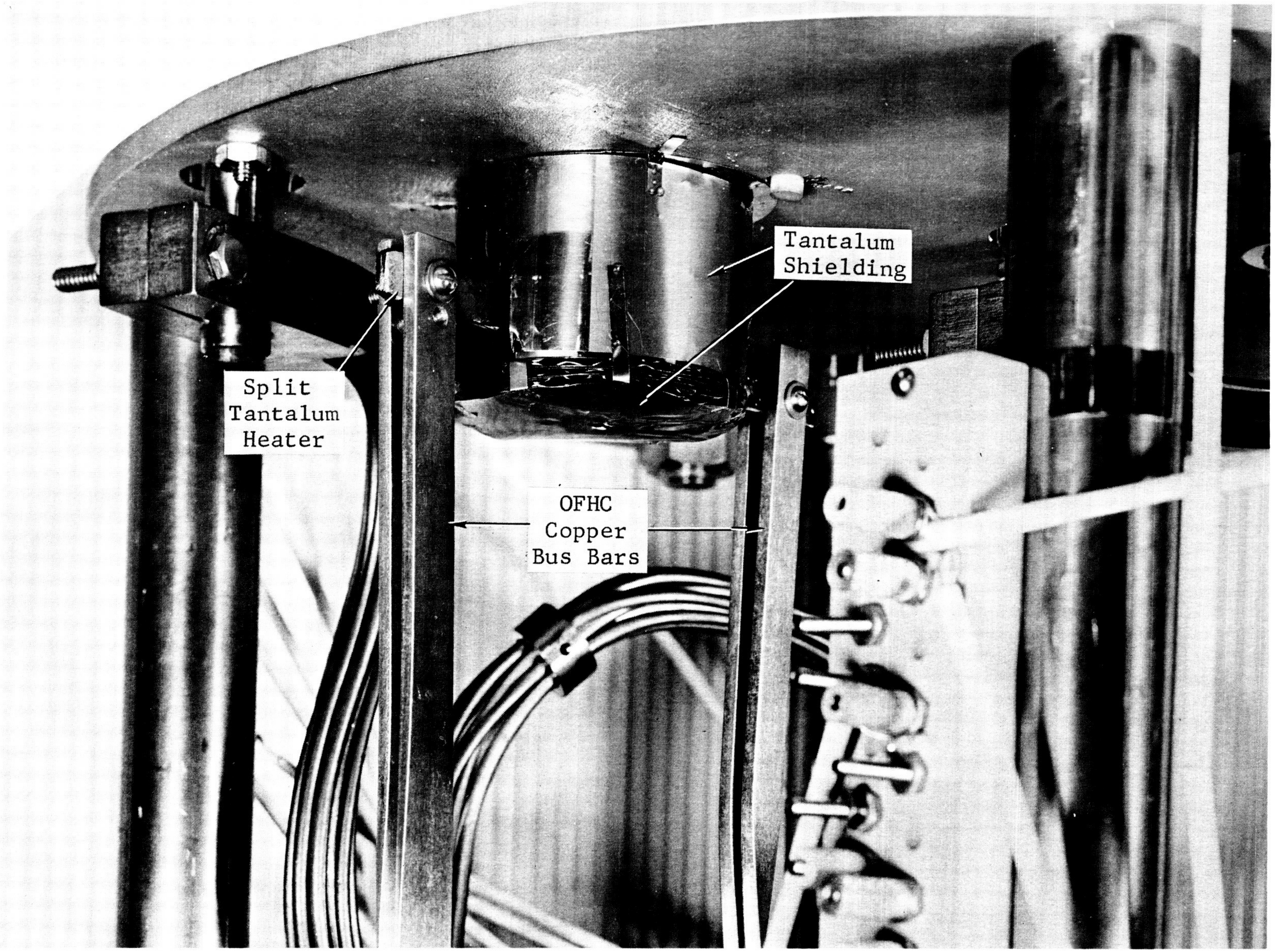

Figure 17. Boiling Nucleator Heater Assembly Installed in Stress Corrosion Test Facility.

calculated from pretest uniaxial creep data. After additional temperature adjustments and creep rate measurements, a temperature of 2100°F was selected to give the desired creep strain (5-10%) in the time duration specified (500-2,000 hours). As of 6-26-65, 430 hours of test time had been accumulated. The creep strain calculated from expansion data in the liquid and condensing zones is shown in Figure 18. Estimated time for 5% creep strain in the liquid zone from this data is 680 hours. The apparent anomally existing between uniaxial creep data and capsule creep data is not understood at this time.

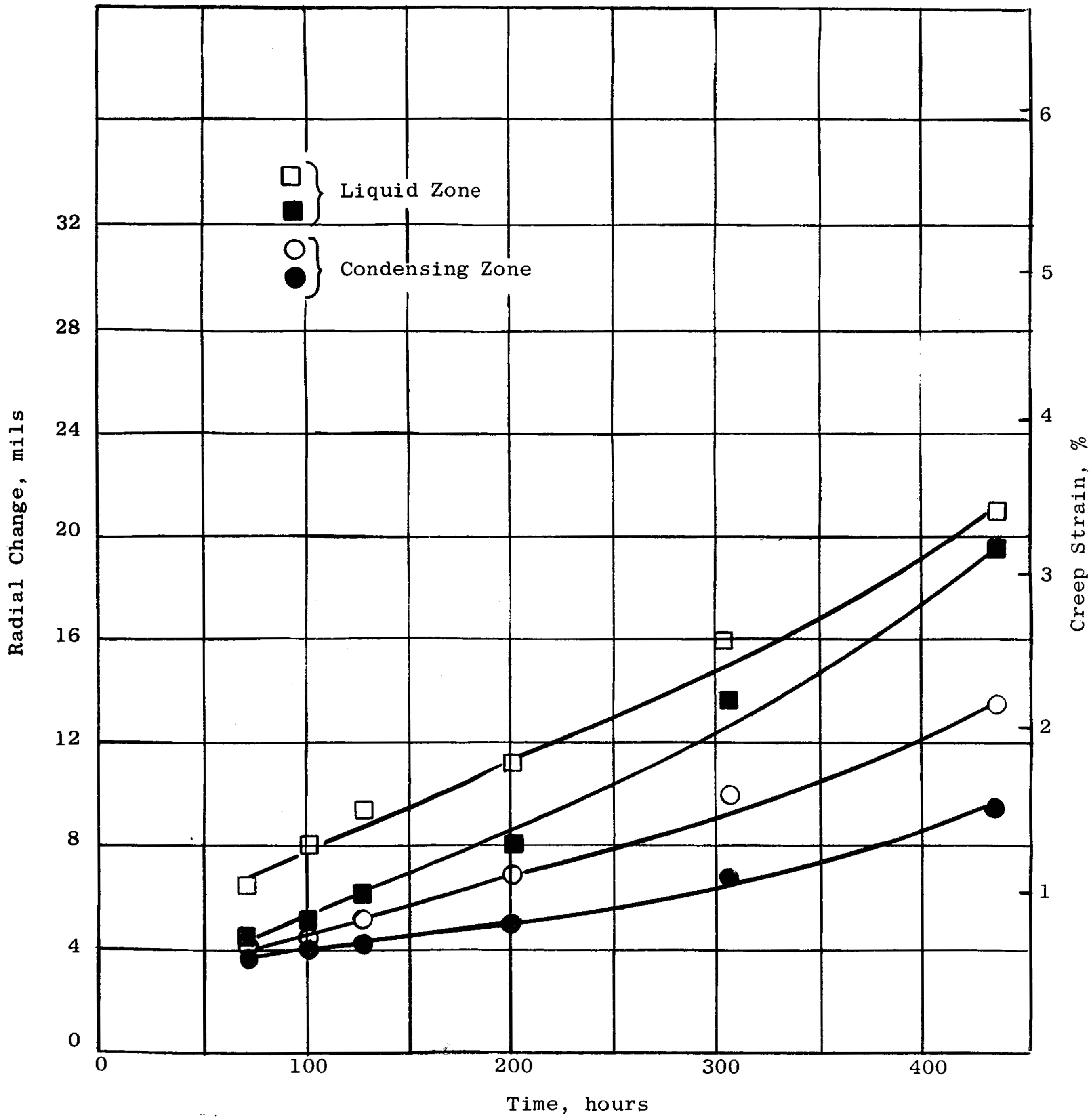

Figure 18. Biaxial Creep Data on the D-43 Stress Corrosion Reflux Capsule Test I as of 430 Hours Exposure to Potassium at 2100°F.

IV. TASK II - BIMETALLIC ISOTHERMAL CAPSULE TESTS

A. Test Evaluation

Metallographic examination of the stainless steel capsules and Cb-1Zr alloy specimens has been completed. Whereas no observable change in metallographic structure was observed in Cb-1Zr alloy specimens tested in Type 321SS capsules, Figure 19, two distinct layers, believed to be CbC and Cb_2N as reported in similar investigations,[2,4] were observed on the Cb-1Zr alloy specimens tested in the Type 316SS, Figure 20. No change in metallographic morphology was observed in the Type 321SS capsule material, Figure 21. However, gross sensitization and sigma phase formation occurred in the Type 316SS, Figure 22. The reduced amount of chromium carbide precipitation and the increased amount of iron-chromium sigma phase formation at the capsule ID results from the depletion of carbon due to the mass transfer reactions. Although the majority of the sigma phase has been pulled out during metallographic preparation of this specimen, some selective removal of this phase at the surface by liquid potassium is possible. Similar microstructural effects were observed in an investigation of Type 316SS boiling potassium test loops at ORNL as described by D. H. Jansen and E. E. Hoffman[5].

Spectrographic analyses of the Cb-1Zr alloy specimens tested in both Type 316SS and Type 321SS capsules showed traces of iron and nickel transferred from the stainless steel to the Cb-1Zr alloy.

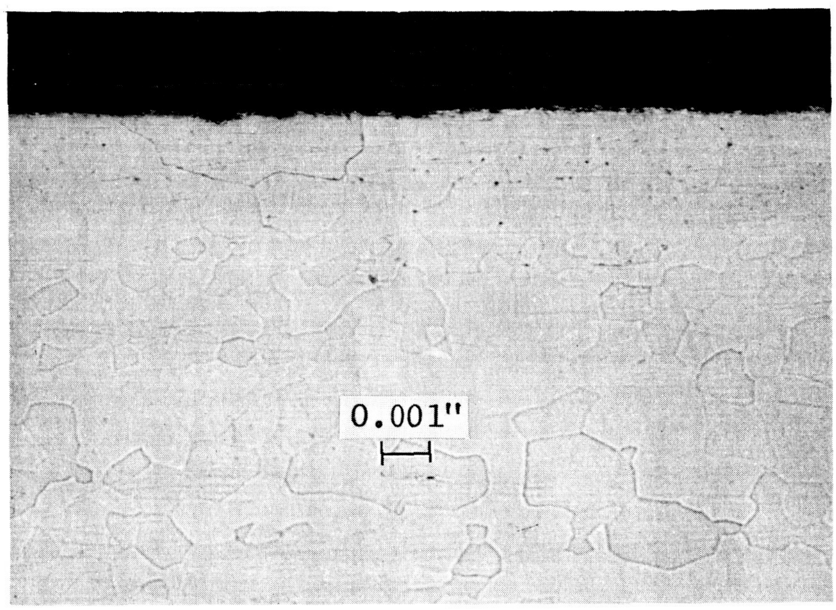

Figure 19. Cb-1Zr Alloy Exposed for 1,000 Hours in Potassium Contained in a Type 321SS Capsule Heated Isothermally at 1400°F.
Etchant: 20% HNO_3 - 20% HF - 60% Clycerol
Mag: 250X

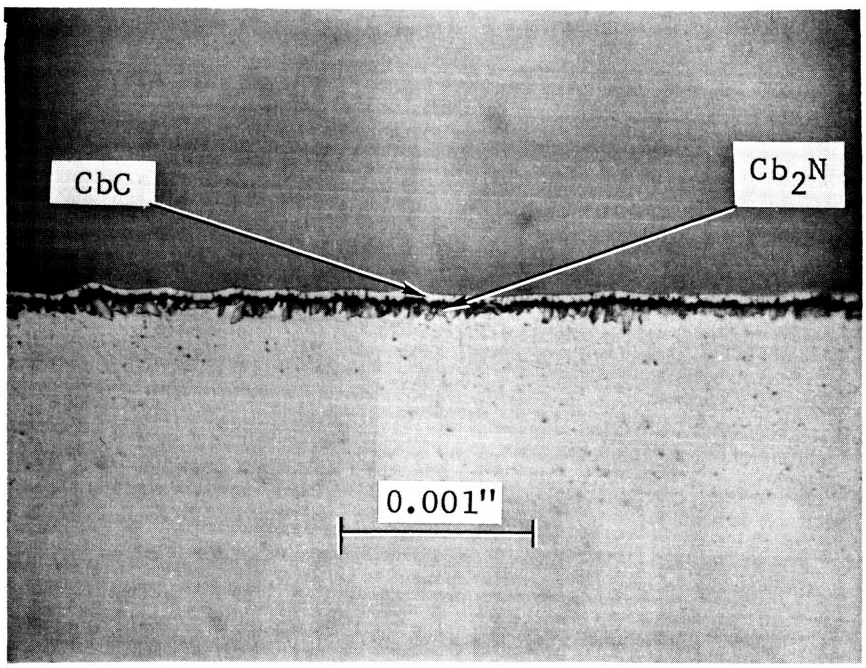

Figure 20. Cb-1Zr Alloy Exposed for 1,000 Hours in Potassium Contained in a Type 316SS Capsule Heated Isothermally at 1400°F Layers Believed to be CbC and Cb_2N.
Etchant: As Polished Mag: 1000X

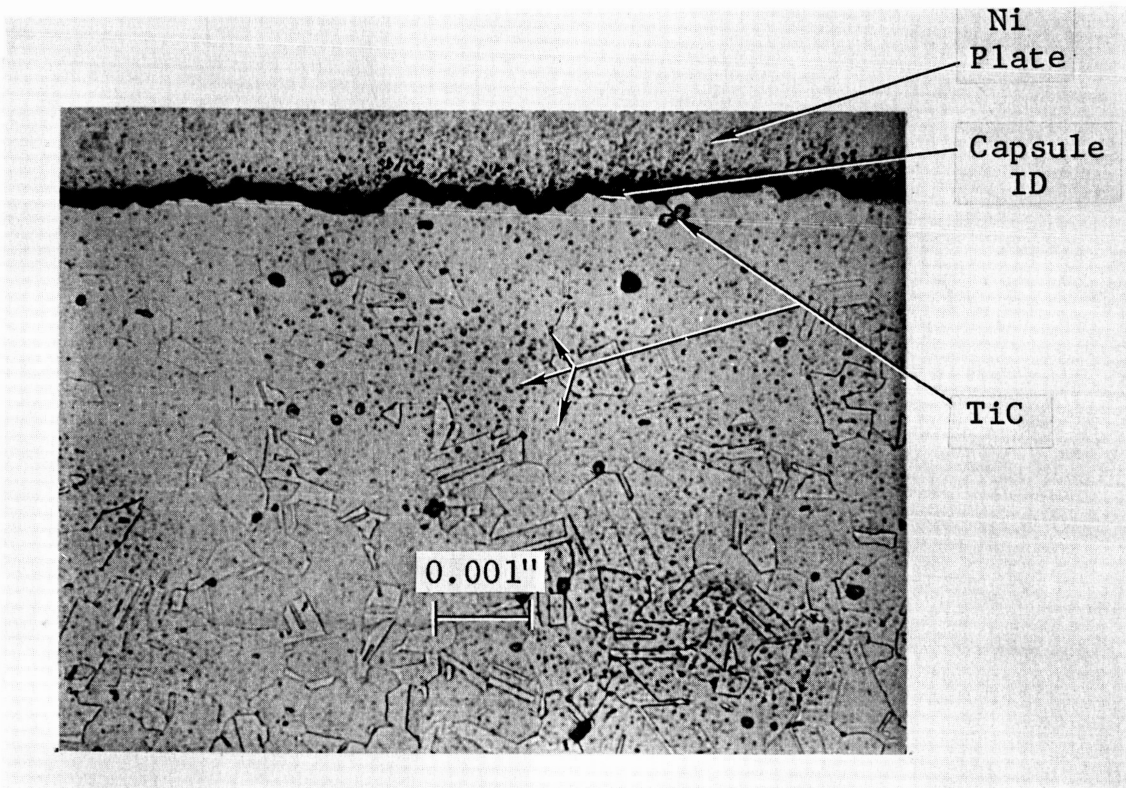

Figure 21. Microstructure of a Type 321SS Capsule After Being Heated Isothermally for 1,000 Hours at 1400°F. The Inner Surface of the Capsule Wall was Exposed to Liquid Potassium.
Etchant: Electrolytic, Oxalic Acid
Mag: 500X

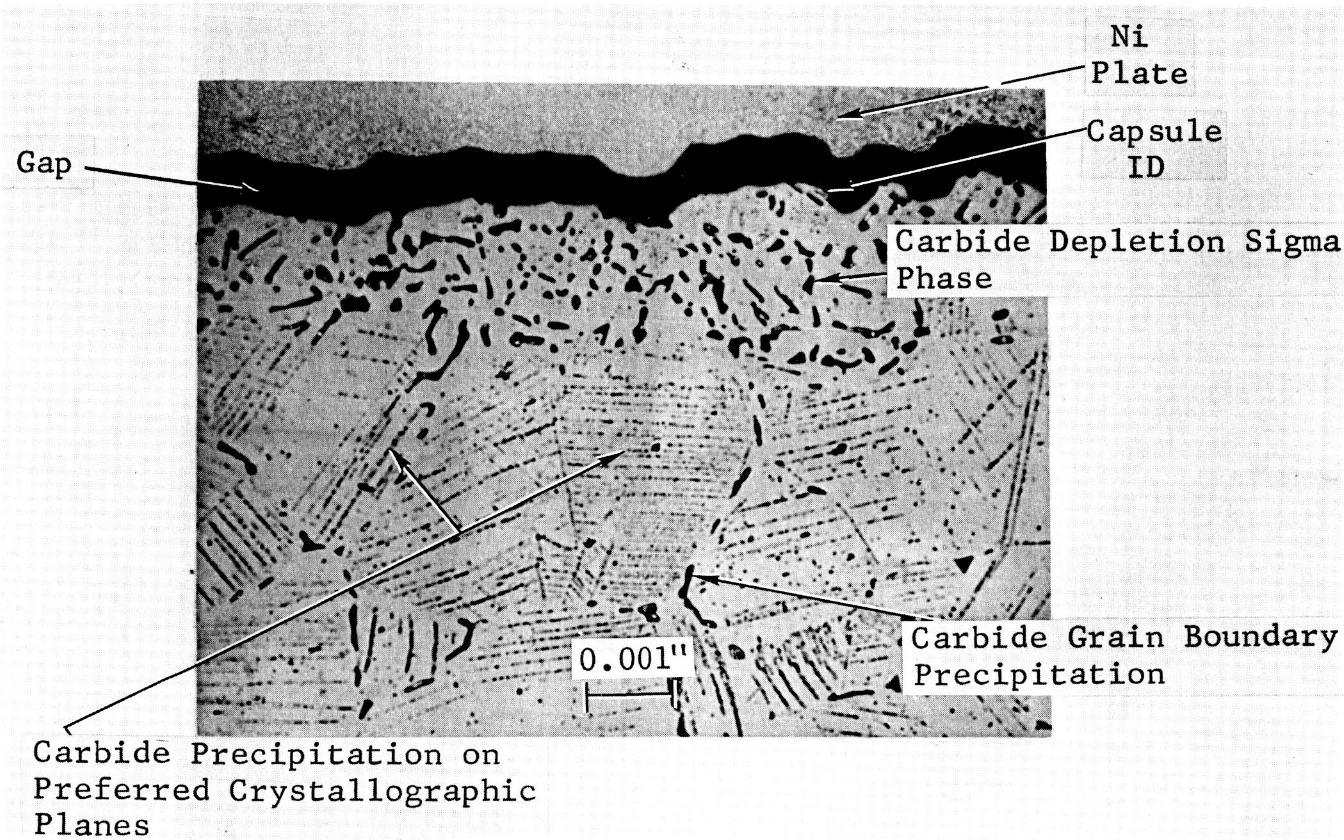

Figure 22. Microstructure of a Type 316SS Capsule After Being Heated Isothermally for 1,000 Hours at 1400°F. The Inner Surface of the Capsule Wall was Exposed to Liquid Potassium.
Etchant: Electrolytic, Oxalic Acid
Mag: 500X

Stress-rupture testing of the 0.040-inch thick Cb-1Zr specimens exposed to potassium in Type 316SS and Type 321SS capsules for 1,000 hours at 1400°F has been completed. The results, Table I, clearly indicate the effects of carbon and nitrogen mass transfer on the properties of Cb-1Zr alloy specimens tested in Type 316SS capsules. Again, little change was noted in the properties of the Cb-1Zr alloy specimens tested in the Type 321SS capsules. The first stress-rupture tests were conducted at 2000°F. At this temperature diffusion of carbon and nitrogen from the carbide-nitride layer, produced during the 1400°F test exposure on the Cb-1Zr alloy in Type 316SS capsules, would accentuate the increased strength. Rupture tests at 1400°F would, therefore, be more meaningful as they would more closely indicate the strength differences resulting from capsule exposure alone. The measured 1400°F-25,000 psi creep rate of the Cb-1Zr alloy specimen which was exposed to potassium in a Type 321SS capsule (Type 321SS-4) indicated an inconveniently long-rupture life. The specimen was, therefore, loaded to 30,000 psi after 259 hours but ruptured on reloading. The specimen exhibited 22% elongation over a 1.25-inch gauge length. A similar test was then performed on a Cb-1Zr alloy specimen exposed to potassium in a Type 316SS capsule, (Type 316SS-2). This specimen ran an additional 100 hours after reloading and the test was terminated without rupture. An elongation of only 2.6% over a 1.25-inch gauge length was measured.

TABLE I

STRESS-RUPTURE PROPERTIES OF 0.040-INCH THICK Cb-1Zr ALLOY SPECIMENS (1) CONTAINED IN TYPE 321SS AND TYPE 316SS CAPSULES AND EXPOSED TO POTASSIUM FOR 1,000 HOURS AT 1400°F

Capsule Material and Specimen No.	Stress psi	Test Temperature, °F	Rupture Life, Hours	Environment Torr
As-Received	10,000	2000	11	7×10^{-8}
Type 316SS-1	10,000	2000	95	5×10^{-8}
Type 321SS-3	10,000	2000	5	7×10^{-8}
Type 321SS-4	25,000	1400	259(2)	6×10^{-9}
	30,000	1400	--(3)	
Type 316SS-2	25,000	1400	260(4)	4×10^{-9}
	30,000	1400	100(5)	

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(1) MCN 454

(2) Specimen Did Not Rupture.

(3) Specimen Failed on Reloading, Total Elongation 22%.

(4) Specimen Did Not Rupture.

(5) Test Terminated Without Rupture, Total Elongation 2.6%.

Post-test evaluation has been completed and has shown the significant advantages of Type 321SS over Type 316SS with respect to carbon and nitrogen transfer in refractory metal-stainless steel-potassium systems. Preparation of a topical report on Task II has begun.

V. FUTURE PLANS

Task I Stress Corrosion Reflux Capsule Program

A. Testing of Capsule Test I will be completed.

B. The capsule will be drained of potassium and cleaned by vacuum distillation. Post-test evaluation will be initiated.

C. Testing of Capsule Test II will commence. Test completion in the next reporting interim is contingent upon the exposure time selected based on results of Capsule Test I.

Task II Bimetallic Capsule Program

A. A topical report will be prepared on this program.

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